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I, SMILJA DRAGOSAVLJEVIC, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2002951194 for a patent by ENVIRONMENTAL SOLUTIONS INTERNATIONAL LTD as filed on 04 September 2002.



WITNESS my hand this
Fourth day of September 2003

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PROVISIONAL SPECIFICATION

Invention Title: Conversion of Sludges and Carbonaceous Materials

The Invention is described in the following statement:

"Conversion of Sludges and Carbonaceous Materials"

Field of the Invention

The present invention relates to the conversion of sludges and carbonaceous materials. More particularly, the present invention relates to a process and apparatus for the production of an improved oil product from the conversion of the organic components of sewage, industrial sludges and other carbonaceous materials.

Background Art

Sludge is the unavoidable by-product of the treatment of sewage and other industrial wastewaters. Traditionally, disposal of such sludge is expensive and typically constitutes half of the total annual costs of wastewater treatment. Historically, the major sludge disposal options have included agricultural utilisation, landfilling and incineration. Also historically, wastewater treatment plants have been designed to minimise sludge production and most effort is expended to stabilise and reduce the sludge volume prior to disposal or utilisation.

The solids component of sewage sludge comprises a mixture of organic materials composed of mostly crude proteins, lipids and carbohydrates. These solids further comprise inorganic materials such as silt, grit, clay and lower levels of heavy metals. For example, a typical raw sewage sludge comprises approximately 50 to 90% volatile matter and 25 to 40% organic carbon. Some sewage sludges already exceed current land application contaminant standards and consequently cannot be used agriculturally or are classified hazardous waste, largely due to their heavy metal and/or organochlorine content.

Many sludge processing options have been proposed in the past. Typically, such options have the potential to convert only a fraction of the organic material into usable energy and very few have been demonstrated as viable net energy

producers at full scale. One example of such processes involves anaerobic digestion of sewage sludge in which approximately 35% of available organic materials is converted to produce a gas rich in methane. Other alternatives have included starved air incineration and gasification.

- 5 A significant problem associated with the above prior art processes relates to the fact that the principle usable energy-containing products are gases which are generally not easily condensable and are of a low net energy content. Accordingly, such gases are impossible or uneconomic to store and must generally be used immediately. Further, it is generally only practicable to use
- 10 them to produce relatively low grade energy, such as steam, and flare them to waste during periods of little or no demand. Not surprisingly, it is preferable that any process used result in storable (liquid or solid), transportable and if possible, upgradeable energy-containing products. Such products would include synthetic oils. It is consequently desirable that there be optimum production of storagable
- 15 energy having any non-storable products, used in the operation of the process itself.

Disposal of sewage sludge has become more problematic recently due to the fact that:

- a) Agricultural use of sewage sludge is restricted by its contaminant content, particularly heavy metals and recently identified endocrine disrupting compounds and other pharmaceuticals,
- 20 b) Ocean disposal is banned,
- c) Landfilling is to shortly be banned in the European Union; and
- 25 d) Incineration of sewage sludge is opposed by the public primarily with respect to the "dioxin issue" (reformation of dioxin during hot flue gas cooling).

In US Patents 4618735 and 4781796, there is described a process and apparatus for the conversion of sludges by heating and chemical reaction in

order to obtain useful storable products therefrom, including oils. The process comprises the steps of heating dried sludge in a zone in the absence of oxygen to a temperature of at least 250°C for the volatilisation of oil producing organic material therein, resulting in heating zone gaseous products and sludge residue,

5 removing the said gaseous product from the heating zone; thereafter contacting heated sludge residue in a reaction zone with the removed heating zone gaseous products in the absence of oxygen at a temperature of 280°C to 600°C for repeated intimate gas/solid contact at temperatures sufficient to cause gas/solid contact, oil producing reactions to occur within the heating zone,

10 gaseous products containing oil products; removing the reaction zone gaseous products from the reaction zone and separating at least the condensable oil products therefrom.

Also disclosed is an apparatus for the conversion of sludge, said apparatus comprising an enclosure establishing a heated heating zone having an inlet thereto for dried sewage sludge and separate outlets therefrom for heating zone gaseous products and residual heating zone solid products; conveyor means within the heating zone enclosure for conveying solid products from its inlet to its solid products outlet; and enclosure establishing a heated reaction zone having separate inlets thereto for gaseous and solid products and separate outlets therefrom for gaseous and solid products; conveyor means within the reaction zone enclosure for conveying solid products from its solid products inlet to its solid products outlet; a heating zone solid products outlet being connected to the reaction zone solid products inlet for the passage of solid products between them; and duct means connecting the heating zone gaseous products outlet to

20 the reaction zone gaseous products inlet.

25

- This apparatus is commonly referred to as a "single reactor" system.

In US 5847248 and 5865956 there is disclosed a process and apparatus based on the process and apparatus of US Patents 4618735 and 4781796, with the following improvements.

The gaseous products from the heating zone are transferred to either an indirect or direct condenser with oil/water separation. The resulting oil and/or non-condensable products are injected into a second reactor. Sludge residue or char from the first reactor is transferred to the second reactor by way of a transfer line.

- 5 The transfer line is equipped with a valve system to ensure that no gaseous products by-pass the condensation system.

In the second reactor provided with heating means, the heated sludge residue from the first reactor is contacted with the revapourised oil or oil and non-condensable gaseous products from the condensation system in the absence of

- 10 oxygen at a maximum temperature of 550°C. Such allows reductive, heterogenic, catalytic gas/solid phase reactions for the generation of clean products and high quality oil product. A conveyor and motor is provided to move the solid product or char through the second reactor.

Gaseous products are subsequently removed from the second reactor for

- 15 passage through a further condenser and oil/water separation system or for ducting to a burner for direct combustion. In the case of passage through a further condenser and oil/water separation system a volume of non-condensable gaseous product, a volume of reaction water and a volume of refined, low viscosity oil is produced. Solid products or char are removed from the second
- 20 reactor by way of a further transfer line having provided therein a screw conveyor for ensuring both no air ingress into and no gaseous product egress from the second reactor. The screw conveyor is connected to a cooling system to cool the solid product or char to less than 100°C before discharge to atmosphere.

- 25 This apparatus is commonly referred to as a "dual reactor" system, be it with or without intermediate oil condensation.

International Patent Application PCT/AU00/00206 describes a process and apparatus for the conversion of carbonaceous materials has as one object thereof to provide a more simple and cost effective process and apparatus still able to provide the various advantages of the process and apparatus of US

- 30 Patents 5847248 and 5865956. This is described as being achieved through

use of a catalytic converter to receive the gaseous product of the first reactor. Gaseous product from the catalytic converter is then condensed to produce water and an oil product. The gaseous product of the reactor may be condensed, thereby separating the oil product from the gaseous products prior to

5 Introduction to the catalytic converter.

The temperature of the catalytic converter is up to 650°C, and preferably in the range of 400 to 420°C, thereby promoting reductive, catalytic gas / solid phase reactions and substantially eliminating hetero-atoms, including nitrogen, oxygen, sulphur, and halogens. The catalytic converter contains a catalyst, the catalyst

10 being chosen from any of zeolite, activated alumina, γ -aluminium oxide, silicon oxide and oxides of alkali, earth alkali and transition metals.

This is commonly referred to as a "Catalytic Converter" system.

Fundamental to each of the systems described previously is a reliance on the fact that vapour and char flowrates are a function of the sludge feed rate ("SFR")

15 and the fraction of sludge vapourised (f). Thus:

$$\text{Vapour Flowrate} = \text{SFR} \cdot f \quad (1)$$

$$\text{Char Flowrate} = \text{SFR} (1 - f) \quad (2)$$

Since all reactors described to date have positive sludge/char conveying systems, the mass of char in the reaction zone is purely a function of char flowrate and the solids retention time (SRT) of the char, which is a function of the screw speed and pitch. The mass of char in the reaction zone is thus:

20

$$\text{Char Mass} = \text{SFR} (1 - f) \cdot \text{SRT} \quad (3)$$

Weight Hour Space Velocity ("WHSV") is a parameter used in the design of some vapour-phased catalytic converter systems. Substituting equations (1) and

25 (3) into the equation for WHSV reveals that:

$$\text{WHSV} = \frac{f}{(1 - f) * \text{SRT}} \quad (4)$$

Consequently, in prior art conversion reactors, the WHSV is purely a function of char SRT, for any given sludge (which defines f).

- 5 This is a major limitation of the prior art since to achieve an acceptable WHSV, very high SRT's, and thus very low conveyor speeds, are required. These low conveyor speeds provide poor mixing of the solid product and hence poor heat and mass transfer in the reactors.

- 10 A further factor apparent in the prior art that needs addressing relates to the presence of free water in the sludge. Typically sludges are commercially dried to between 10 and 5% water. In the conversion reactors this water flashes to steam, with a significant volume increase, which reduces the residence time of the oil vapours in the reactor. It would thus be advantageous, for sludges with more than 5% water, to remove this water by heating to about 105°C, prior to entry to the conversion reactors.
- 15

It is one object of the present invention to provide a process and apparatus for the conversion of sludges and carbonaceous materials that allow adequate mixing of solid product and thereby provides an acceptable WHSV when compared with the processes and apparatus of the prior art.

- 20 The preceding discussion of the background art is intended to facilitate an understanding of the present invention only. It should be appreciated that the discussion is not an acknowledgement or admission that any of the material referred to was part of the common general knowledge in Australia as at the priority date of the application.
- 25 Throughout the specification, unless the context requires otherwise, the word "comprise" or variations such as "comprises" or "comprising", will be understood

to imply the inclusion of a stated integer or group of integers but not the exclusion of any other integer or group of integers.

Disclosure of the Invention

In accordance with the present invention there is provided a process for the 5 conversion of sludges and carbonaceous materials, the process comprising the steps of:

- 10 (a) Heating the material to be converted in a heating zone of a reactor in the absence of oxygen for the volatilisation of oil producing vapours, thereby producing both a vapour product and a solid residue or char;
- 15 (b) Contacting the vapour product and char in a reaction zone of the reactor at a determined Weight Hour Space Velocity ("WHSV") so as to promote vapour-phase catalytic reactions; and
- (c) Removing and separating the gaseous products and char from the reactor.

Preferably, the gaseous products from the reactor may be condensed to produce oil and water. The oil and water may then be separated and the oil polished to remove char fines and any free water.

Still preferably, the inventory of char within the reactor is able to be adjusted to 20 provide the required WHSV in the reaction zone of the reactor.

The dried sludge and char are fed to, and removed from the reactor by a means to ensure no ingress of air into the reactor, or egress of vapours from the reactor.

The temperature of the reactor is preferably at least 250°C. The temperature of the reactor is still preferably 400 to 450°C.

The process of the present invention may further comprise the additional step of drying the material to be converted to less than 5% moisture prior to introduction to the reactor.

In accordance with the present invention there is further provided an apparatus

5 for the conversion of sludges and carbonaceous materials, the apparatus comprising a reactor having a heating zone and a reaction zone and a means for conveying the material through both zones of the reactor in turn, the heating zone having a material inlet and the reaction zone having a material outlet and a gaseous product outlet, wherein there is further provided a means for retaining

10 the material within the reactor such that a desired Weight Hour Space Velocity ("WHSV") for the material is achieved.

Preferably, the means for conveying material through the reactor is not one that may be described as a positive conveyance means, for example a screw conveyor. The conveying means is preferably one that allows a level of back

15 mixing of the material being conveyed.

Brief Description of the Drawings

The present invention will now be described, by way of example only, with reference to one embodiment thereof and the accompanying drawings, in which:-

20 Figure 1 is a block diagram describing a process for the conversion of sludges and carbonaceous materials in accordance with the present invention;

Figure 2 is a cross-sectional side view of an apparatus for the conversion of sludges and carbonaceous materials in accordance with the present invention;

25 Figure 3 is a cross-sectional end view of the apparatus of Figure 2 taken along line A thereof;

Figure 4 is a cross-sectional end view of the apparatus of Figure 2 taken along line B thereof;

Figure 5 is a graph showing a plot of oil viscosity against WHSV demonstrating the correlation therebetween.

5 Best Mode for Carrying Out the Invention

A significant amount of operational data and experience has been generated by the applicants with regard to the thermal conversion of sludges, using the processes and equipment of the prior art as described hereinabove.

The "single reactor" system described hereinabove has been tested/demonstrated using continuous pilot plants, operating at scales of 1 and 10 kg/h. The "dual reactor" system described hereinabove has been tested/demonstrated, operating in both intermediate condensation (IC) and non IC modes, using continuous pilot plants operating at scales of 1 and 20 kg/h. A full-scale commercial plant, designed on the "dual reactor" basis, has been operated at sludge throughputs of up to 800 kg/h. The "catalytic converter" system described hereinabove has been tested/operated using a continuous pilot plant, operating at throughputs of up to 1 kg/h.

The commercial plant was designed and built as a dual reactor system primarily due to mechanical constraints in building a single reactor of this size. It was however, believed that the dual reactor system had other advantages, particularly the ability to easily have different solids retention times in the two reactors, which serve different functions. In addition, whilst the commercial plant was designed to operate with IC, operational issues precluded this mode of operation and the plant operated without IC.

After intensive review and interpretation of the operational data from all of these facilities it became apparent to the applicants that the quality of the oil produced was exclusively a function of operating temperature and the Weight Hour Space Velocity ("WHSV") achieved in the second reactor (or the reaction zone of single

reactor systems). As noted hereinabove, the WHSV is a parameter used for the design of vapour-phased catalytic converter systems.

The WHSV is defined as the mass flow rate of the vapours to be converted divided by the mass of the catalyst in contact with the vapours. For the sludge 5 conversion systems described above, the WHSV is thus:

Mass Flowrate of Vapours (kg/h) in Second Reactor (or Reaction Zone of Single Reactor Systems)
Mass of Char (kg) in Second Reactor (or Reaction Zone of Single Reactor Systems)

Oil viscosity data as a function of WHSV, from three different conversion systems, using two different sludges is shown in Figure 5. As can be seen, there 10 is a very good correlation between oil viscosity and WHSV, using reactors operating at throughputs from 1 kg to 800 kg/h. The data in Figure 5 was obtained at an operating temperature of 400°C. Figure 5 confirms clearly that WHSV is the parameter which controls oil viscosity, irrespective of sludge type or reactor configuration.

15 The process and apparatus of the present invention will now be described by way of reference to a single embodiment thereof. The example is not to be considered as limiting, but rather is simply one example of how both the process and apparatus of the present invention may be implemented.

In Figure 1 there is shown in block diagram the process of the present invention.

20 Material to be converted, for example dry sludge with a total solids ("TS") of greater than 80% may be fed to an additional drying step prior to introduction to a reactor. It is envisaged that materials to be converted that contain greater than 5% water will be subjected to additional drying, with the water subsequently removed being passed to a waste water treatment plant of known type.

25 The reactor, in accordance with the present invention, to which the material to be converted is passed will be described hereinafter with reference to Figures 2 to 4. The char produced through the heating and reaction of the material to be converted within the reactor is passed from the reactor to a char cooler after which it may be reused in the process of the present invention.

Vapour produced from the material to be converted may be passed directly to combustion or may alternatively be directed to a condenser after which the oil and water produced may be separated and the oil polished to remove char fines and any free water. The oil thus produced may be passed to reuse. Non-condensed gases from the condenser may be passed to reuse, as may be reaction water obtained from the oil/water separation step.

In Figure 2 there is shown an apparatus 10 for the conversion of sludges and carbonations materials comprising a reactor 12 and a means for conveying material through the reactor, for example a conveyor 14.

- 10 The apparatus 10 further comprises a sludge feed hopper 16, the base of which is provided with a screw conveyor 18 arranged to pass sludge to a sludge inlet 20 through which sludge may be passed into the reactor 12. Still further, the reactor 12 has provided therein a gaseous product outlet 22 and a material outlet 24. Char may pass from the reactor 12 to a char hopper 26, the char hopper 26 15 in turn being provided with a screw conveyor 28.

Additionally, the reactor 12 is provided with a heating means (not shown) and a coating of thermal insulation 30.

- 20 The reactor 12 is functionally divided into two zones, a heating zone 32 and a reaction zone 34. As sludge is passed through the inlet 20 into the reactor 12 it is conveyed from the inlet 20 through the heating zone 32 by the conveyor 14. The sludge is heated in the absence of oxygen for the volatilisation of oil producing vapours in the heating zone 32. This produces both the oil producing vapours and a solid residue, referred to as the "char". The conveyor 14 conveys the char from the heating zone 32 and through the reaction zone 34 towards the 25 outlet 24 and simultaneously promotes interaction of the vapours with the char so as to promote vapour-phase catalytic reactions in the reaction zone 34.

The conveying means 14 comprises a drive 36, a shaft 38 and a bearing 40 supporting the shaft 38. Within the reactor 12, the shaft 38 is provided with paddles 42 or the like which allow a level of back-mixing. It is envisaged that the

levels of back-mixing promoted within the heating zone 32 and the reaction zone 34 may be different. However, the governing factor for determining the retention time within the reactor is the desired weight hour space velocity ("WHSV") and the WHSV of Weight Hour Space Velocity.

- 5 So as to facilitate the retention of the char within the reactor to achieve the desired WHSV, a means for retaining the char is provided in the form of a weir 44. The weir 44 is provided immediately before the char outlet 24. The weir 44 is provided as an adjustable-height weir such that the height of the weir 44 may be altered to achieve the desired WHSV in the reaction zone 34.
- 10 The conveyor 14 is envisaged to specifically not comprise a "positive conveyance" screw conveyor. It is further envisaged that the rotational speed is to be at least 1 rpm. Further, the heating rate within the heating zone 32 is envisaged to be between 5 and 30°C/min.

In summary, the operational experience described above in accordance with the present invention has indicated the benefit to be gained by changing the design of the reactors to eliminate the dependency of char inventory on SRT, i.e. to decouple the influence of screw speed on inventory and using the WHSV as the sole basis for the mass transfer design of the reactor.

- 20 Apparatus designed in accordance with the present invention is envisaged to overcome the limitations of the prior art systems and it is further envisaged that a single reactor will be able to handle throughputs of greater than 25 dry tpd of high VS sludge, since it would be much smaller and manufacturing constraints would also be eliminated.

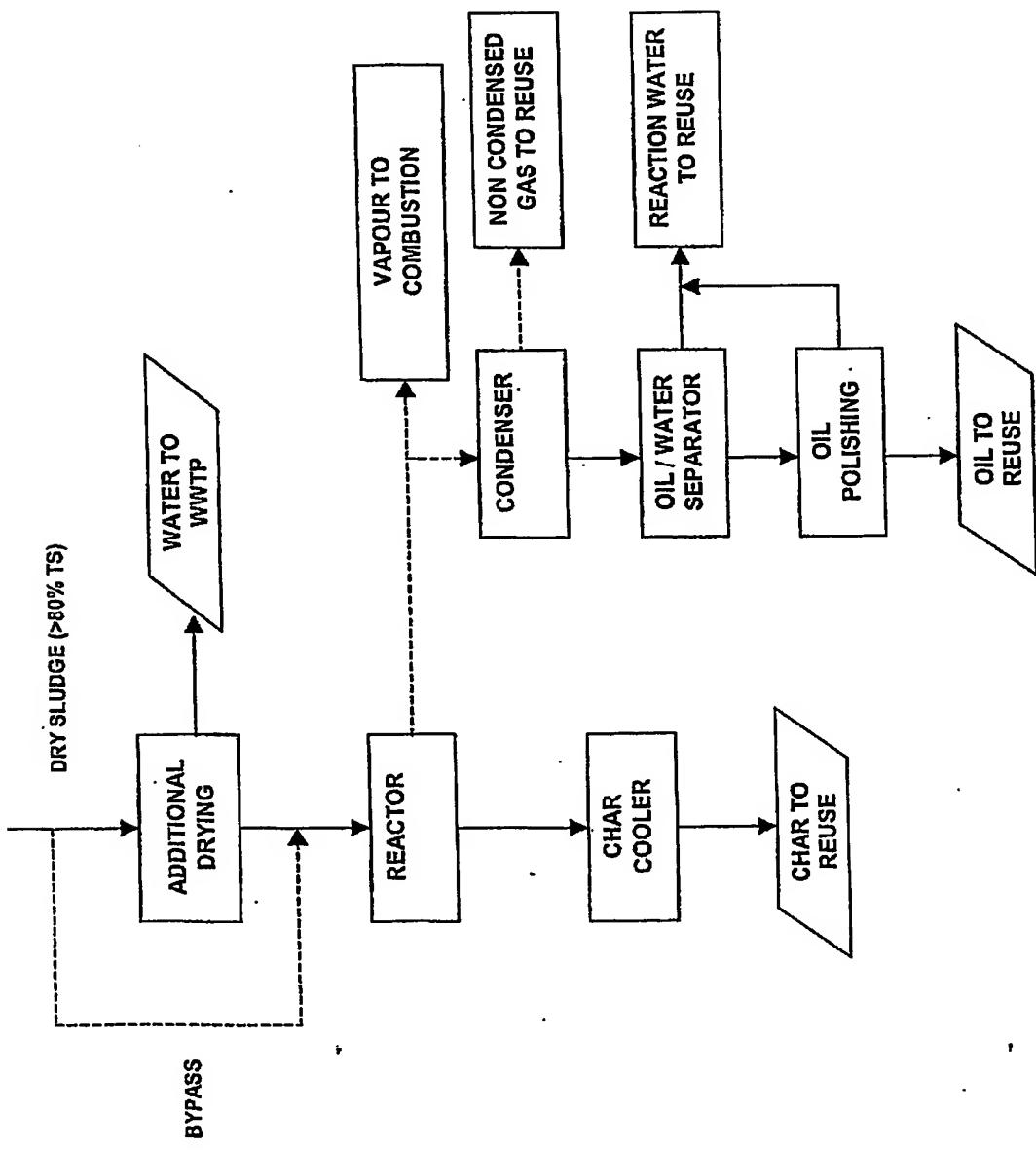
Modifications and variations such as would be apparent to the skilled addressee are considered to fall within the scope of the present invention.

Dated this Fourth day of September 2002.

Environmental Solutions International Ltd
Applicant

Wray & Associates
Perth, Western Australia
Patent Attorneys for the Applicant

Figure 1



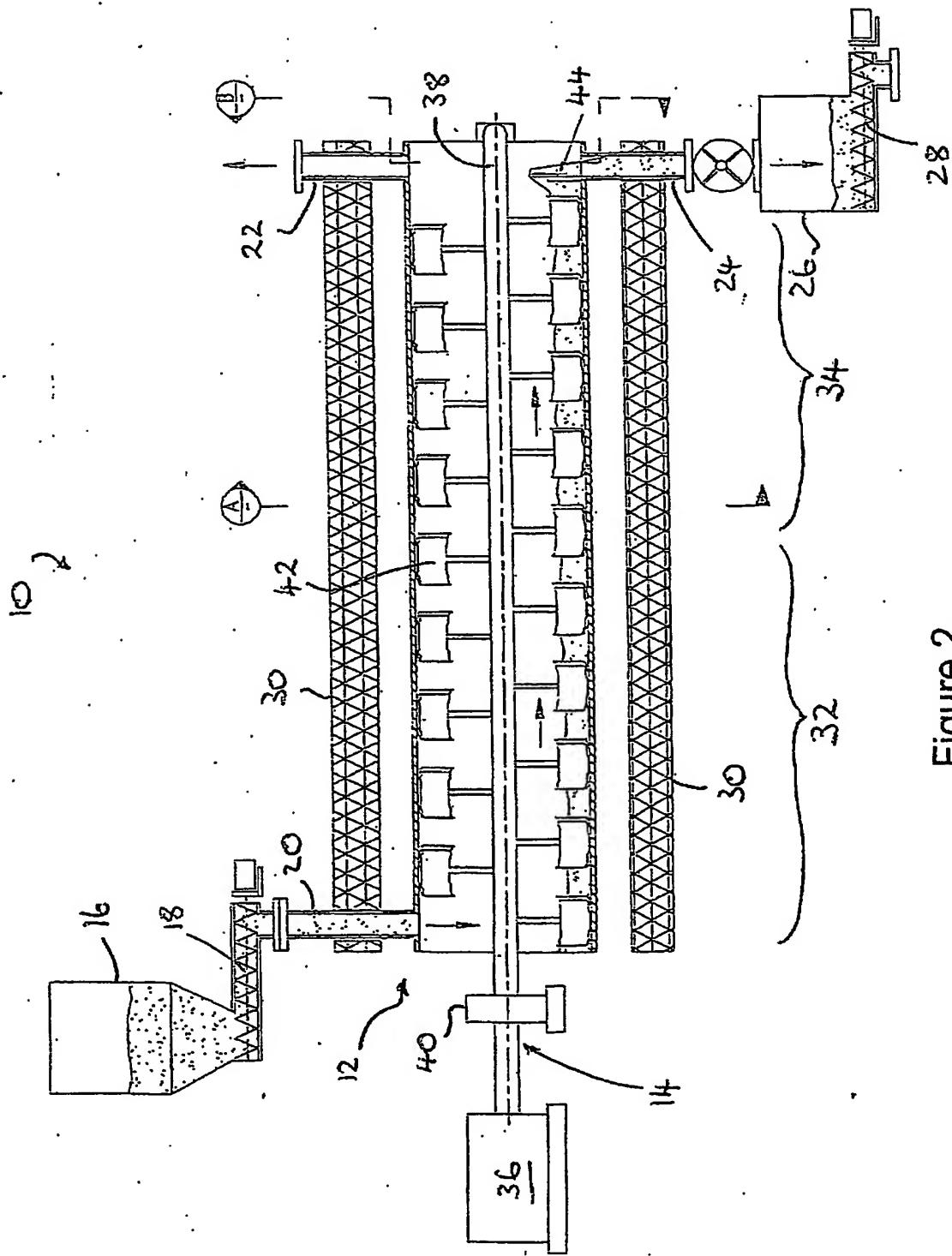


Figure 2

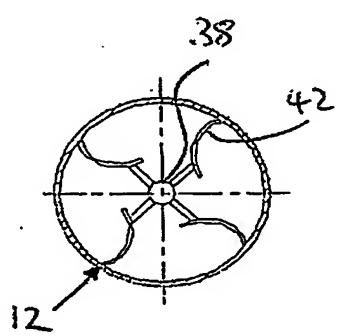


Figure 3

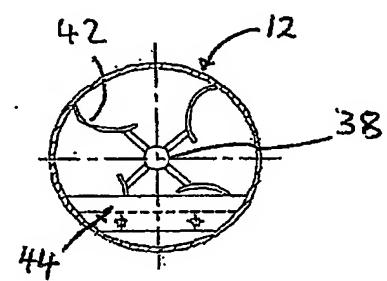


Figure 4

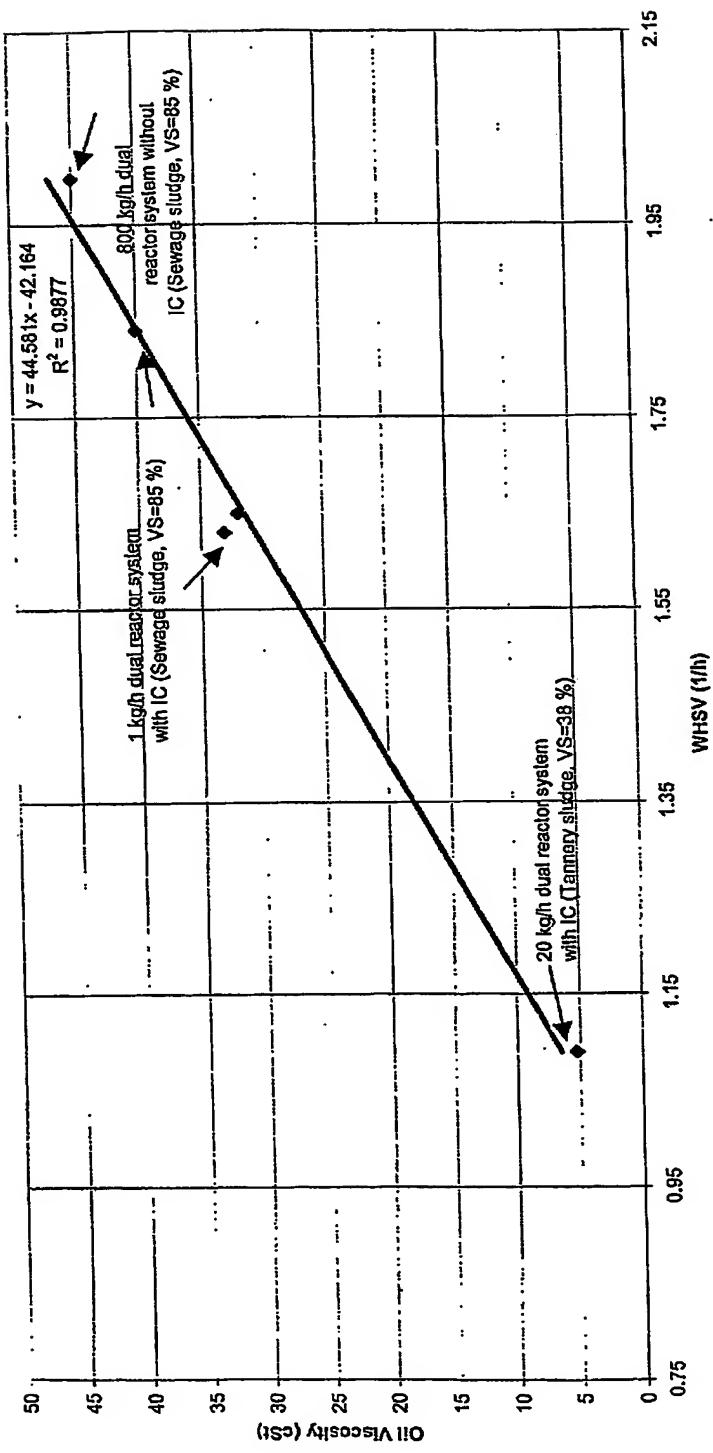


Figure 5

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